# A YBCO RF-SQUID VARIABLE TEMPERATURE SUSCEPTOMETER AND ITS APPLICATIONS

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#### **ABSTRACT**

The SQUID susceptibility using a high-temperature rf SQUID and a normal metal pick-up coil is employed in testing weak magnetization of the sample. The magnetic moment resolution of the device is  $1 \times 10^{-6}$  emu, and that of the susceptibility is  $5 \times 10^{-6}$  emu/cm<sup>3</sup>.

### 1. INTRODUCTION

Since Colcough et al. (1) first revealed the existence of intrinsic rf SQUID effect in high-T<sub>c</sub> superconducting Y-Ba-Cu-O, Zimmerman et al. (2) are the first who deviate from the applicable high temperature rf SQUID. Since then, many laboratories, including ours, have made rf SQUIDs with their own features (3-8). Before a liquid nitrogen superconducting flux transformer can be made, a pick-up coil made of normal metal is used in this device. The intention of this work is to substitute the low-temperature liquid helium device with the high-temperature SQUID while keeping the extraordinary features of a SQUID variable temperature susceptometer (VTS).

The temperature range of the VTS developed in this laboratory is from 77 K to 300 K, and the magnetic field range, 0-0.1T. The device has been used to characterize superconducting thin films.

#### 2. EXPERIMENTAL SETUP AND OPERATIONS

The VTS includes the SQUID system, sample chamber, magnet, dewar, and a computerized data acquisition system. The SQUID system consists of the SQUID sensor, signal pick-up coil and correspondent electronics. The SQUID used here is a double-hole rf SQUID made of a YBCO bulk material working at 19 MHz. The flux resolution at 77 K is  $5 \times 10^{-4} \, \Phi_o / v$ Hz at the frequency range of 20-200 Hz, and  $2 \times 10^{-4} \, \Phi_o / v$ Hz near dc end.

Figure 1 gives the noise spectrum of the device at 77 K. The inset is its noise output characteristics near dc end. To reduce the external interferences, the SQUID is placed in a YBCO superconducting screening hollow cylinder. In this experiment, a pick-up coil made of normal metal is used to substitute the superconducting coil before a satisfactory superconducting flux transformer working in liquid nitrogen can be made. The inductances of the pick-up coil and the input coil do not have to be matched in the 77 K SQUID. When a signal is picked up with a normal metal coil, the amount of the signal depends not only on the moving speed of a sample, but also on its relative position. If a pick-up signal is calibrated with a standard sample in the same way that a sample is measured, then a normal metal pick-up coil can also generate a signal propor-

tional to magnetic moment. This idea is the mail feature of this apparatus, which bursts the idea that a superconducting flux transformer must be used in the application of a SQUID. Our recent test of a superconducting flux closed circuited in liquid nitrogen temperature using the SQUID

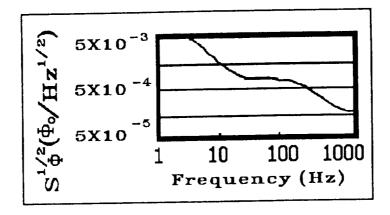


Figure 1. The noise spectrum of the device at 77 K.

shows that a closed flux transformer can be made (9), although it is not yet mature enough to be used in a susceptometer. The normal metal coils used in this experiment are a pair of counterwound coils 100mm apart from each other and with the same number (N=60) of rings on each coil. The electronics employed here are those used in a usual liquid helium rf SQUID similar to the S.H.E. 330 SQUID system. Our sample chamber system consists of a thermostat, a temperature measuring and controlling part, and a sample transmission mechanism. The temperature difference between the sample space and the sample itself is corrected by the calibration.

A normal metal solenoid is used to supply the magnetic field for the system before a satisfactory high-T<sub>c</sub> superconducting magnet can be made. The normal magnet used here supplies a center field as high as 0.1 T when the current is 2 A.

The vacuum adiabatic method is used to keep constant temperature. The temperature decrease is obtained with a phosphorous-copper ring mounted between the sample space and the liquid nitrogen chamber. The sample, which is suspended on a string, enters the sample space from the top of the dewar. When a heater is not turned on, the typical liquid nitrogen consumption is about 2 liters/day.

The procedures of sample measuring are similar to those for the liquid helium SQUID susceptometer. The difference in measuring with a superconducting flux transformer is that with a susceptometer, the amount of the signal has to be calibrated using a standard sample while maintaining the moving sample's orientation. The standard samples are annealed high-purity aluminum and gallium cylinders.

## 3. EXPERIMENTAL RESULTS

The experimental results demonstrate that the design of the normal metal coils does work, except the absolute value of the susceptibility has to be calibrated.

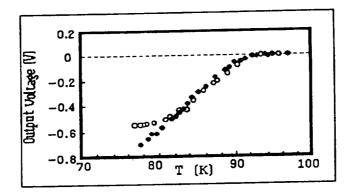


Figure 2. Meissner and screening effects of a small sample of YBCO.

Figure 2 displays the Meissner and magnetic screening effects of a small piece of Y-Ba-Cu-O sample (3 mg) detected by this device when the external field is 100 Oe. In measuring the magnetic screening effect, the magnetic field is lowered to zero first; the sample temperature is then dropped to 77 K from room temperature. After a certain magnetic field is applied, magnetic moment signal is measured at increasing temperatures. In measuring Meissner effect, the magnetic moment signals are measured at a given field while the sample temperature is lowering from a temperature well above T<sub>c</sub>. The results in Figure 2 show that the Meissner signal is about 60 percent of that of screening effect, which gives useful information for improving the sample making technology.

Figure 3 gives a magnetic transition curve of a YBCO superconducting thin film detected by this apparatus. The film thickness is about 3000 to 5000 A, and the applied field is 3 Oe. The transition temperature determined by this VTS is 82.3 K which is very close to the value  $T_{co}$ , zero-resistance temperature found by the four-probe method. In the case when the SQUID is functioning as a sensor, the measured field is, in principle, a relative change, and a calibration has to be done to determine the absolute value of moment and susceptibility from a signal--even in a liquid helium SQUID susceptometer.

It is more important when normal metal pick-up coils are used in the liquid nitrogen device. A special moment calibrating coil is employed to supply a certain value of a magnetic moment. The moment, m, of the coils calculated as m=NSI, where N is the number of the circles of the coil, S is the average area of the coil, and I is the current through the coil. In this experiment, N=30, the average diameter of the coil is about 0.3 cm, and the moment resolution of the device is about  $1\times10^{-6}$  emu due to the signal size and the device resolution. Some metallic diamagnetic materials with high purity, such as aluminum and gallium are used to calibrate the susceptibility values.

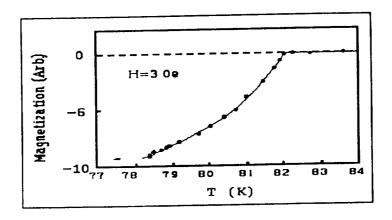


Figure 3. Magnetic transition curve of a YBCO thin film detected by the device.

The calibration shows that the susceptibility resolution of the device is consistent with that estimated from the measurement on a small sample of high- $T_c$  superconductor, namely, about  $5x10^{-6}$  emu/cm<sup>3</sup>.

In summary, the SQUID susceptibility using a high-temperature rf SQUID and a normal metal pick-up coil can be employed in testing weak magnetization of a sample. The susceptibility transition curve of superconducting thin film and the calibrations of metallic diamagnetic samples show the feasibility of usage of the non-superconducting transformer on the SQUID. The magnetic moment resolution of the device is  $1 \times 10^{-6}$  emu, and that of the susceptibility is  $5 \times 10^{-6}$  emu/cm<sup>3</sup>.

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